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Synthesis of Nickel Oxide / Cerium Dioxide/ Graphene Oxide (NiO/CeO_2/GO) Composite as an Organic Pollutant Remover of Dyes

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ABSTRACT

Before industrial effluents are released into the environment, several techniques have been developed to remove colours from them. These techniques include ion exchange, photocatalytic degradation, chemical oxidation, adsorption, and osmosis. Since these dyes may be totally broken down by photo-catalytically active semiconductors when exposed to light, photocatalysis is one of these approaches that is thought to be environmentally friendly and has been widely employed for environmental cleanup In the current study, a hydrothermal approach was used to create a NiO/CeO_2/GO nanocomposite that was heated for 24 hours at 180 °C using a Teflon liner. To confirm the creation of nanocomposite material, the synthesised nanomaterials were characterised using a variety of spectroscopic methods, including XRD, FTIR, Raman Spectroscopy (RS), SEM, Photo Luminescence Spectra (PL), and Dynamic Light Scattering (DLS). The materials that were synthesised were investigated as photocatalysts for the breakdown of Methylene Blue (MB) dye when exposed to sunlight. The product's photocatalytic activity on methylene blue was examined. It has been demonstrated that adding GO increases absorption while lowering bandgap energy. The elimination effectiveness of methylene blue from an aqueous solution was investigated, and it was found to be 93.22% when the ideal circumstances of NiO/CeO_2/GO of 0.10 mg/L, pH 5, and contact duration of 40 min under visible light were used.

دراسة أزالة المركب النانوي (أكسيد النيكل \ ثاني أكسيد السيريوم \ أكسيد الغرافين) للملوثات العضوية في الأصباغ

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الكلمات المفتاحية:

المواد المركبة
الأصباغ
الميثيلين الأزرق
الملوثات العضوية
الجرافين
التقنية الحرارية المائية

الملـخـص:

تم تصميم عدة طرق لإزالة الأصباغ من النفايات الصناعية السائلة قبل تصريفها في البيئة. وتشمل هذه الأساليب الأكسدة الكيميائية ، والتبادل الأيوني والتحلل الضوئي والامتزاز والتناضح، يعتبر التحفيز الضوئي تقنية خضراء وقد تم استخدامه على نطاق واسع للعلاج البيئي حيث يمكن أن تتحلل هذه الأصباغ تماما بواسطة أشباه الموصلات النشطة ضوئيا تحت إشعاع الضوء. في هذه الدراسة ، تم تصنيع مركب النانو (أكسيد النيكل / ثاني أكسيد السيريوم/ أكسيد الغرافين) وتسخينها في درجة حرارة 180 درجة مئوية ولمدة 24 ساعة. وقد تم دراسة مركب النانو بواسطة وتحويل فوريه للطيف بالأشعة تحت الحمراء ومطيافية رامان وصورة طيف الأنبعاث و تشتت مختلف التقنيات الطيفية ، مثل حيود الأشعة السينية الضوء الديناميكي وذلك لتأكيد تكون المواد النانوية. تم دراسة المواد المركبة كمحفزات ضوئية لتحلل صبغة الميثيلين الأزرق تحت أشعة الشمس. تم تحليل النشاط التحفيزي الضوئي للمركب النانوي على الميثيلين الأزرق. وقد تبين أن دمج أكسيد الغرافين يعزز الامتصاص ، ويقلل أيضا من فجوة الطاقة. وقد تم اختيار الميثيلين الأزرق كمادة عضوية شائعة للتحقيق في كفاءة الأزالة من المحلول المائي ، والتي بلغت 93.22 ٪ في حين كان مركب النانو 0.10 ملغ/لتر بدرجة حموضة 5 ولفترة 40 دقيقة تحت الضوء المرئي.

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1. Background

Dyes have been widely employed in a variety of industries, including textile, tanning, cosmetics, and pigment [1]. Particularly in the textile industry, effluents contain high levels of color compounds that have the potential to seriously pollute the water supply. Additionally, an extensive spectrum of industrial applications frequently uses organic dyes. Therefore, before releasing wastewater into the environment, it is crucial to minimize the dye content. Large volumes of colors, organics, bleach, and salts discharged into water sources can alter the physical and chemical characteristics of fresh water. Dyes in wastewater that are extremely visible, may block light, are stable to heat and light irradiation, and are hazardous to microorganisms. Because of the structure and synthetic origins of dyes, their removal is a particularly complicated operation [2].

Metallic nanoparticles are known to have a variety of physical and chemical characteristics that enable researchers to use them in a wide range of fields, including electronics, biosensors, food, textile, healthcare, the environment, and agriculture [3]. Due to their catalytic activity, metallic nanoparticles have attracted a lot of attention lately for their use in the breakdown of harmful dyes [4]. It is possible to create nanoparticles for the catalytic degradation of textile dyes via a variety of physical, chemical, and biological mechanisms [5].

Numerous contaminants, including dye, heavy metals, antibiotics, and pesticides, are present in the wastewater produced by different sectors. Toxic chemical dyes are one of the main pollutants found in the effluent produced by the textile, plastic, paper, food, tanneries, and pharmaceutical sectors, together with other toxic chemical components. Some of the most common dyes used by industries to add color are azo, basic, acidic, and cationic dyes. However, the nature of these synthetic colors makes them extremely poisonous, carcinogenic, and mutagenic. Wastewater containing dye is immediately discharged by businesses into nearby waterways like rivers, lakes, and drains. Additionally, wastewater produced by various companies is occasionally used directly for irrigational reasons, which degrades the quality of the soil and crops. The presence of dye molecules in the water is poisonous, and they also reduce the amount of sunlight that reaches the water bodies, which has an impact on aquatic life. Therefore, it is crucial to properly cleanse wastewater that contains color before discharging it into nearby bodies of water. Due to its chemical structure, which gives the dye molecule a high level of stability, degradation of the dye molecule to a harmless component is challenging [6].

Large volumes of dye compounds are present in the effluents from the textile sector, which might seriously pollute the water supply. Additionally, an extensive spectrum of industrial applications frequently uses organic dyes. Therefore, before wastewater is released into the environment, it is crucial to lower the dye content. Large-scale discharges of organics, bleaches, and salts can alter the physical and chemical characteristics of fresh water. Dyes in wastewater that are extremely visible, may block light, are stable to heat and light irradiation, and are hazardous to microorganisms. Because of the structure and synthetic origins of dyes, their removal is a particularly complicated operation [7].

Dye degradation is a chemical process that alters a dye's color. This method is frequently used to turn plant extracts into natural colors. Synthetic organic dye is the most popular method for dying textiles. Petroleum products are used to make these colors [8].

The fundamental issue with synthetic colors is that they cannot be broken down by nature. This implies that they have a lengthy lifespan and can survive in the environment. One method for dissolving these synthetic colors is dye degradation. This will make it simpler to get rid of them from the surroundings [9]. Materials known as nanocomposites contain nanoscale particles within a

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matrix of conventional material. When nanoparticles are added, qualities including mechanical strength, toughness, and electrical or thermal conductivity significantly enhance. Since the nanoparticles are so efficient, only 0.5 to 5% of the original material is typically added [10].

A solid compound called a nanocomposite is made up of many layers, at least one of which has one, two, or three dimensions and is as thin as a nanometer [11]. The atoms of the constituent materials are organized as clusters or tiny grains in nanocomposites. Nanocomposites are solid multiphase materials with one of the 1, 2, or 3 phases smaller than 100 nm [12].

Carbon atoms are densely packed into a hexagonal honeycomb lattice to form a single layer, or monolayer, to form graphene. It is an allotrope of carbon with a molecular bond length of 0.142 nanometers that takes the shape of a plane of SP2-bonded atoms. Graphite, which has an interplanar spacing of 0.335 nanometers, is made up of layers of graphene stacked on top of one another. Van der Waals forces, which can be overcome during exfoliation of graphene from graphite, hold the various layers of graphene in graphite together [13].

The term "hydrothermal synthesis" describes the process of creating chemicals by chemical reactions in a sealed, heated solution that is above the surrounding pressure and temperature. Mineral solubility in hot water under high pressure is necessary for the hydrothermal production of single crystals. The crystal development is carried out in a device called an autoclave, which consists of a steel pressure vessel and is supplied with water and a nutrient. The growing chamber is kept at a gradient of temperature so that the seeds develop more at the colder end and the nutrition is dissolved at the hotter end [14].

2. Literature Review

Before industrial effluents are released into the environment, several techniques have been developed to remove colors from them. These techniques include ion exchange, photocatalytic degradation, chemical oxidation, adsorption, and osmosis. Since these dyes can be totally destroyed by photo-catalytically active semiconductors when exposed to light, photocatalysis is one of these approaches and has been widely employed for environmental rehabilitation.

In 2022, K. Subashini, et. al., NiO@GO nanocomposite's photocatalytic performance on the degradation of Methylene blue (MB) and Malachite Green (MG) has been evaluated; the results show that the GO plays a crucial role in the improvement of photocatalytic performance [15].

In 2021, Shu Ting Deng, et. al., when ciprofloxacin was examined for its increased degradation using a cerium oxide/graphene oxide nanocomposite under ultrasonic irradiation, the results showed that after five cycles, the degradation efficiency had essentially not changed. Consequently, the CeO_2/GO nanocomposite could be employed as an environment-safe, recyclable, and cost-effective degrading material for popular antibiotics [16].

In 2020, Somayeh Mohamadi and Mehrdad Ghorbanali, results of the investigation into the adsorption and UV-assisted photodegradation of methylene blue by a graphene sponge decorated with CeO_2 demonstrated that the charge transfer between the CeO_2 and surrounding graphene as well as the cationic methylene blue adsorption on the negatively charged surface of the graphene increase the photocatalytic activity of the nano-composite [17].

In 2018, Jahangir Ahmada, et. al., results of a study on the improved visible light-driven photocatalytic activity of CdO-Graphene oxide heterostructures for the degradation of organic pollutants showed that the cubic structured NiO and GO in NiO/GO nanocomposites create a p-n heterojunction. Additionally, the photocatalytic activity of the NiO/GO nanocomposites was substantially higher than that of the as-prepared cubic NiO nano-powder for the degradation of Methylene blue (MB), which may be related to the p-n heterojunction that formed in the NiO/GO nanocomposites. The best catalytic activity was particularly demonstrated by the p-type NiO/n-type GO nanocomposites with 65% GO. The photocatalyst dissolved about 97% of the initial MB dyes in just 50 minutes [18].

3. Materials and Methodology

3.1 Synthesis of Graphene Oxide

A process that was improved and only slightly modified

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was used to create graphene oxide from natural graphite powder. In general, a flask was constructed with a 9:1 (v/v) mixture of H_2SO_4 and H_3O_3 concentrations. A uniform solid combination of 0.3 g of graphite and 1.8 g of $KMnO_4$ that was kept in a water bath received a slow addition of this concentrated acid mixture. The graphite's oxidation process was then completed and the reaction mixture turned dark brown after being stirred for 12 hours at 50 °C. After the reaction mixture had reached room temperature, 500 mL of ice-cold water was added. After adding a 30% H_2O_2 solution gradually until the reaction mixture's color turned brilliant yellow, the oxidation process was stopped. The solid material was washed with water, 30% HCl, and then ethanol many times as part of the workup process after the filtrate was centrifuged. Vacuum drying the dark-brown crystalline powder of graphene oxide (GO) at room temperature and storing it in a desiccator were the next steps [19]. Figure 1 shows the synthesis of graphene oxide.

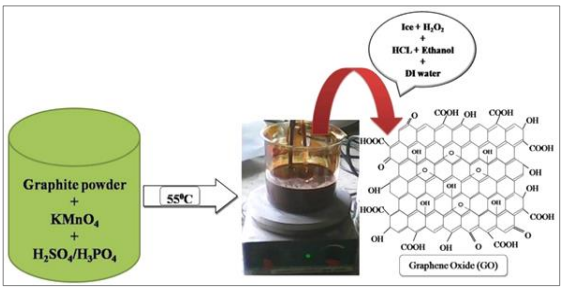

Figure 1: Graphical Illustration of the Synthesis of GO Sheets.

3.2 Synthesis of NiO/CeO_2/GO Nanoparticles

First, using Hummer's process, GO was created from natural graphite powder. GO was dissolved in 100 mL of distilled water using an ultrasonic equipment for 30 minutes. Second, 0.3 M of $Ni(NO_3)_2$ and 0.3 M of $CeCl_3$ were dissolved in 60 mL of distilled water and stirred for an hour. After that, the former was gently added to the latter while stirring and left for a half-hour. Finally, NaOH was added dropwise until the pH was 7 and the mixture was complete. The mixture was placed into a hydrothermal Teflon liner and heated for 24 hours at 180 C. The finished product was repeatedly cleaned with distilled water and ethanol before being dried for eight hours at 60 degrees Celsius in a vacuum oven [20].

4. Results and Discussion

4.1 Crystallite Size Using Scherrer's Method

According to Figure 2, the (100) reflection plane corresponds to the GO peak at about 2 =12.8°. In the NiO/CeO_2 XRD pattern. The diffraction peaks at 2 values of 28.58°, 33°, 47.3°, 56.3°, 69.4°, 76.7°, and 78.7°, respectively, correspond to the crystal planes (111), (200), (220), (311), (400), (411), and (420). which demonstrated the crystalline NiO/CeO_2 synthesis. Additionally, the NiO/CeO_2 diffraction peaks were distinct and strong, which points to the extremely crystalline structure of these nanoparticles. Using the Debye-Scherrer equation, the crystallite size was determined [21]:

$D = K \lambda / \beta \cos \theta$, where D is the crystallite size, K is the form factor (0.9), is the utilized x-ray wavelength (1.5406 nm), is the full width at half maximum (FWHM), and is the Bragg's angle diffraction. The equation determined the average particle size of NiO/CeO_2/GO to be 164 nm. The XRD patterns of the composite NiO/CeO_2/GO are shown in Figure 2.

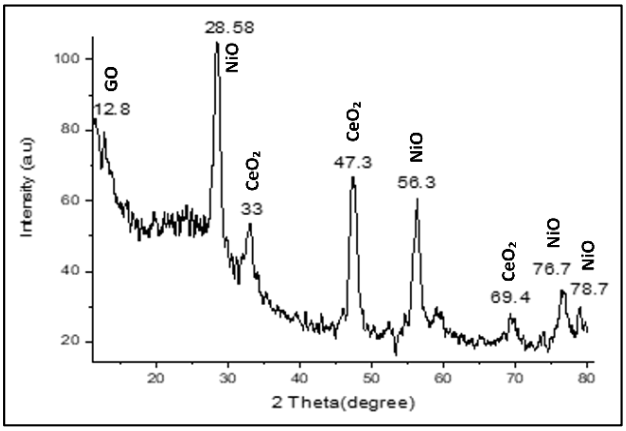

Figure 2: XRD Patterns of NiO/CeO_2/GO Composite.

3.3 Functional Group Analysis (FTIR)

The produced NiO/CeO_2/GO NPs' functional group and chemical composition were located using the FT-IR spectra. Figure 3 displays the NiO/CeO_2/GO NPs' FT-IR spectra in the 600–4000 cm^{-1} range, confirming the presence of several oxygen functional groups such hydroxyl, carboxyl, and epoxy. While 2974 cm^{-1} and 2896 cm1 were assigned to the methyl C-H asymmetric stretching and methyl C-H symmetric stretching,

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respectively, the broadband at 3356 cm^{-1} was related to the stretching band of the hydroxyl group, or OH. The weak band, which appears at 1658 cm^{-1}, is caused by the hydroxyl group's H-O-H bending vibration as a result of chemically adsorbed water molecules. The band detected at 1087 cm1 and 1045 cm^{-1} correspond to epoxy C-O-C stretching vibrations and C-O bending stretching, respectively. The band located at 1381 cm^{-1} can be attributed to C-O stretching vibration. Figure 3 displays the NiO/CeO_2/GO NPs' FTIR spectra from 600 to 4000 cm^{-1}.

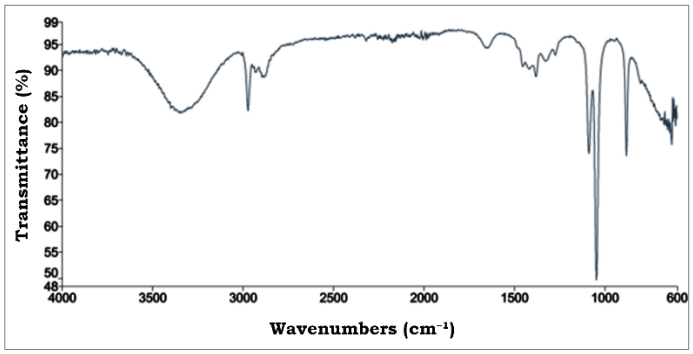

Figure 3: The FTIR Spectrums of NiO/CeO_2/GO NPs in the Range of 600 to 4000 cm^{-1}.

4.4 Raman Spectroscopy

Raman spectroscopy, which is regarded as a potent technique for the characterization of carbon-based materials, was used to further clarify the structure of the NiO/CeO_2/GO nanocomposites and the electronic interactions between graphene oxide and NiO/CeO_2. Raman spectroscopy is a useful tool for differentiating between the ordered and disordered crystal structures of carbon and is highly dependent on electrical structure. As-made NiO/CeO_2/GO nanocomposites' Raman spectra were captured in the 400–4000 cm^{-1} spectral region.

The trembling peaks of metal oxides are responsible for the weak peak at 462 cm1. The D band and G band of graphene are shown by the peaks at 1347 cm1 and 1617 cm1, respectively. While the D band is associated with the defect and lattice distortion of carbon and peaks at 2959 is the second-order two phonon process, the G band is a doubly degenerate phonon mode (E2g symmetry) at the Brillouin zone (BZ) center that is Raman active for SP2 carbon networks. The degree of the samples' defects can be determined using the ID/IG ratio, which measures the intensity of the D to G band. A higher ratio indicates a larger fault. The composite NiO/CeO_2/GO Raman spectra are shown in Figure 4.

4.5 SEM Analysis

The spherical shape of the NiO/CeO_2 nanoparticles is highly ornamented on the surface of wrinkled graphene oxide sheets, as seen in the SEM image of the NiO/CeO_2/GO nanocomposite. It is further shown that the NiO/CeO_2 nanoparticles and GO sheets develop a sufficient interfacial contact. Effective charge transfer between NiO/CeO_2 nanoparticles and GO sheets is made possible by this form of interfacial contact between metal particles and GO sheets. Figure 5 displays the nanocomposite of NiO, CeO_2, and GO after SEM inspection.

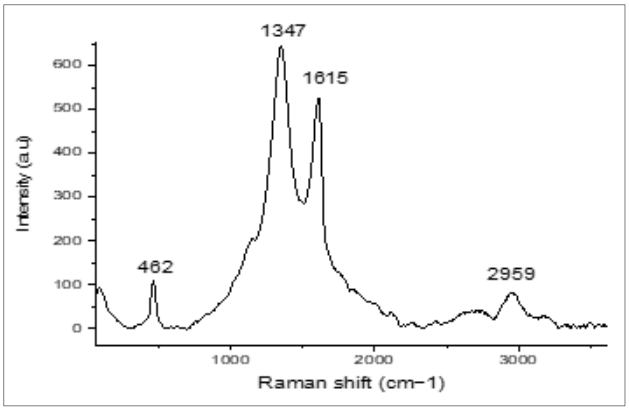

Figure 4: The Raman Spectra of NiO/CeO_2/GO Composite

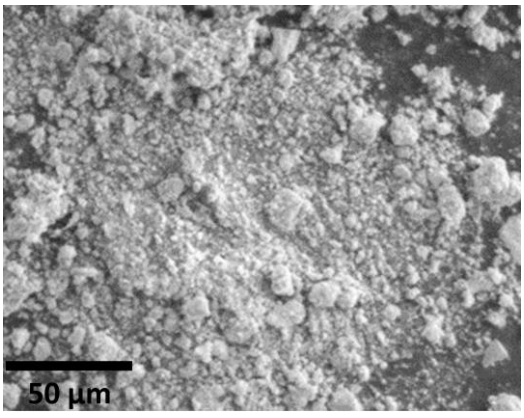

Figure 5: The Image of SEM Analysis of NiO/CeO_2/GO Nanocomposite.

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4.6 Photo-Luminescence (PL)

The purpose of the PL experiment was to confirm in more detail how well the photogenerated electrons and holes were separated in the NiO/CeO_2/GO nanocomposite. The emission centering at 360 nm in the PL spectrum is related to deep level emissions, which typically develop as a result of surface flaws. Additionally, the emission peaks at 420 and 465 nm can be attributed to surface state emission or deep trap emission and band edge or near band edge emission, respectively. Additionally, the peaks at 500 and 535 nm are attributed to vacancies, point defects that can easily form recombination centers and are either intrinsic or extrinsic. The photo-luminescence spectra of NiO/CeO_2/GO are shown in Figure 6.

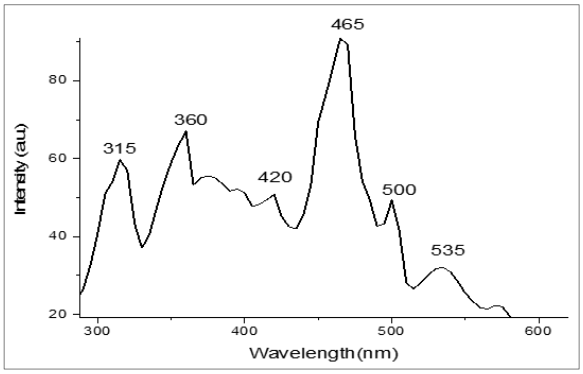

Figure 6: The Photo-Luminescence spectra of NiO/CeO_2/GO.

4.7 Dynamic Light Scattering (DLS)

The NiO/CeO_2/GO histogram of DLS particles is produced by the variance in scattered light produced by Brownian motion using randomized mobility of the particle in a solvent. These particles were spread more widely and clumped together to form a larger sphere, which had an average diameter of 900–920 nm. The NiO/CeO_2/GO DLS particles histogram is shown in Figure 7.

4.8 UV-Vis Spectra

Because NiO/CeO_2/GO nanoparticles could be photoexcited and produce more electron-hole pairings, the UV-V is spectra are important. This may increase photocatalytic efficiency when exposed to light waves. The following equation, which is based on the inter-band theory, can be used to determine the band gap values from the Tauc plot: $(\alpha h_v)^n = (h_v - E_g)$, where denotes the absorbance factor, h the Planck's constant, v the photon frequency (Hz, 1/s), and E_g the energy band gap (eV). Numerous different electrical transitions are related to the constant known as the coefficient n. Interesting NiO/CeO_2/GO nanoparticles with a 2.1 eV band gap range and interesting electrical characteristics. The Tauc plots of the NiO/CeO_2/GO absorption spectra are shown in Figure 8.

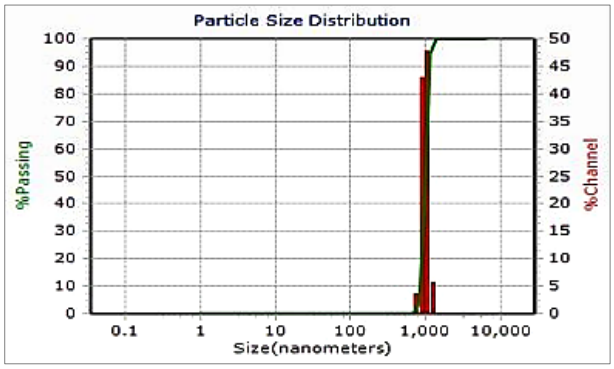

Figure 7: The DLS Particles Histogram of NiO/CeO_2/GO.

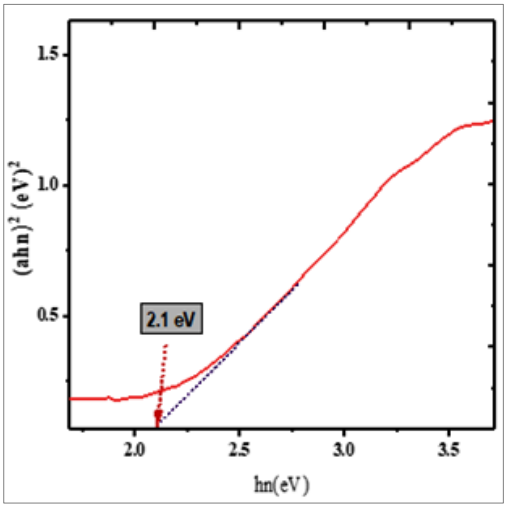

Figure 8: The Tauc Plots of the Absorption Spectra of NiO/CeO_2/GO.

4.9 Photo-Catalytic Degradation of MB Dye for Different Sample Concentration

The NiO/CeO_2/GO nanoparticles were shown to be rather robust under visible light photocatalysis in the present investigation, but the MB dye rapidly degraded (93.22%) after 40 minutes of exposure to natural sunshine. A pH range of 2 to 7 was used to investigate the photocatalytic efficacy; the most effective pH was chosen for further research as pH 5.

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The link between adsorption and photodegradation in solid-liquid systems can be described by a pseudo-first order kinetic model, and experimental data were fitted using the rate equation: - ln (C_t/C_o) = $k_{(p)}$. t, where, Ct is the absorbance of MB at t time; C_o is the absorbance of MB after dark adsorption; t is the irradiation time; and $k_{(p)}$ is the pragmatic kinetic constant. Figure 10 shows the photocatalytic activity of NiO/CeO_2/GO towards Methylene blue. Figure 11 shows the photocatalytic degradation of methyl orange by different samples and Figure 12 shows the - ln (C_t/C_o) versus time and photocatalytic reaction rate constants. The photo-catalytic degradation percentage and the first order kinetics of NiO/CeO_2/GO composite samples are shown in Table 3.

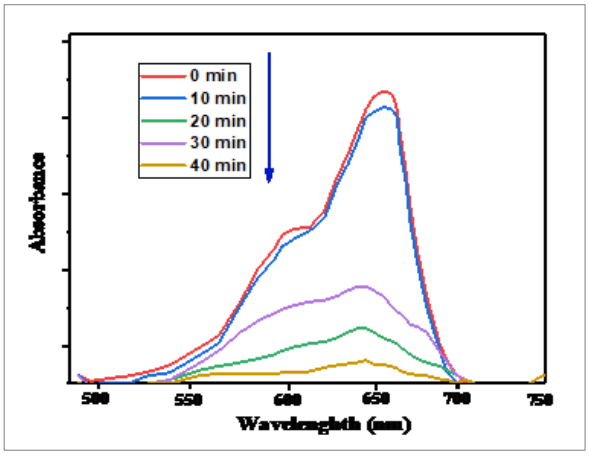

Figure 10: The Photocatalytic Activity of NiO/CeO_2/GO towards Methylene Blue.

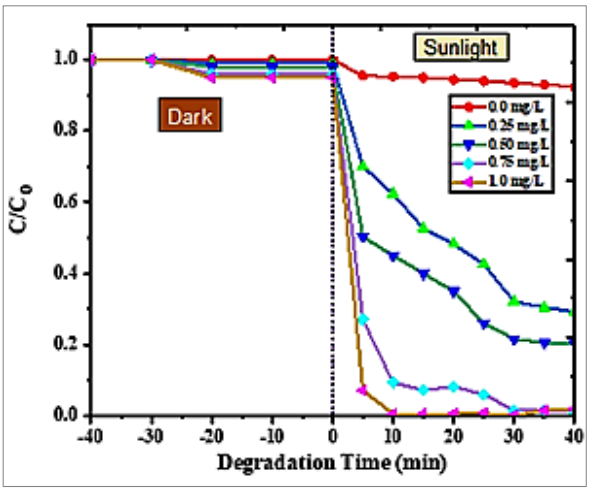

Figure 11: The Photocatalytic Degradation of Methyl Orange by Different Samples.

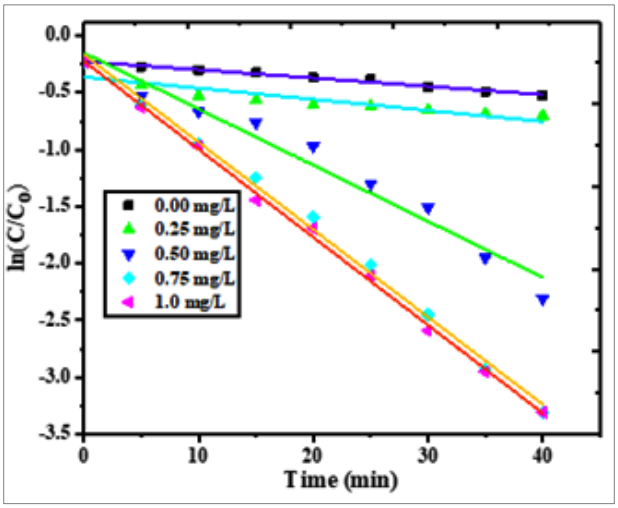

Figure 12: The -ln (C_t/C_o) Versus Time and Photocatalytic Reaction Rate Constants (in the insect).

Table 3: The Photo-Catalytic Degradation Percentage and the First order Kinetics of NiO/CeO_2/GO Composite Samples

S. No	Sample (mg/L)	Photocatalytic Degradation (%)	First Order Kinetics (K) min^{-1}
1	0.25	53.23	0.132
2	0.50	76.56	0.262
3	0.75	92.18	0.422
4	0.10	93.22	0.428

4.10 Reusability

The NiO/CeO_2/GO nanoparticles' capacity to breakdown MB dye repeatedly depends on their sustainability. The final byproducts of the breakdown of MB dye are carbon dioxide and water. A significant reduction in MB, photodegradation was observed as illustrated in Figure 13. Six cycles of MB dye degradation were performed using same NiO/CeO_2/GO nanoparticles, all of which had notable effect. After each cycle, the photocatalyst was removed, cleaned with distilled water and methanol, and then left to dry overnight before being used again. With a gradual decline in photocatalytic activity percentages of 89.24%, 86.36%, 83.31%, 82.5%, and 81.8%, respectively, up to the fifth cycle for MB degradation, loss of catalytic recyclability was observed.

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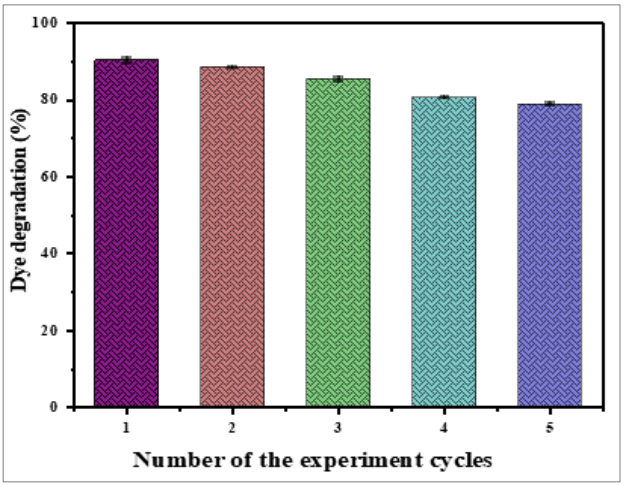

Figure 13: Recyclability of Photocatalytic Activities of $NiO/CeO_2/GO$ for Five Successive Cycles.

5. Conclusions

In conclusion, a $NiO/CeO_2/GO$ nanocomposite was created using a hydrothermal method. Product's photocatalytic activity on methylene blue was examined. It has been demonstrated that adding GO increases absorption while lowering bandgap energy. The elimination effectiveness of methylene blue from an aqueous solution was investigated, and it was found to be 93.22% when the ideal circumstances of $NiO/CeO_2/GO$ of 0.10 mg/L, pH 5, and contact duration of 40 minutes under visible light were used.

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